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(72) Inventors HANS-GEORG FITZKY and
HORST LIMP

(54) PROCESS FOR THE CONTINUOUS DETERMINATION OF THE WATER CONTENT OF WEBS

(71) We, BAYER AKTIENGESELLSCHAFT, 509 Leverkusen, Germany, a body corporate organised under the laws of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the continuous determination of the water content of a web by measuring its microwave absorption. This process is particularly suitable for determining small quantities of water in webs of paper or foil, e.g. photographic films or papers.

In the industrial production of these materials and in particular in automated production, a rapidly responding and simply operating measuring device is required for determining the water content. A device operating without contact with the material may be used for measuring rapidly moving webs, or a device which contacts the sample may be used for solving laboratory problems, as required.

Thus, for example, the residual moisture of photographic paper and film must be kept within certain limits in order to prevent electrostatic charging during the photographic process and to prevent any change in the rolled film or paper due to excessively high water content.

Compared with infrared reflection or transmission measurement which have frequently been used up to now, the microwave measuring process has the advantage that the water measurement is not interfered with by OH-groups in other structures, by powerful scattering additives or by insufficient optical transparency in the region of the 1.9 μm band. Thus, for example, paper coated with heavy spare or paper layers several millimetres in thickness can easily be tested for their water content. Another advantage is the property of microwave transmission measurement of measuring the water content of the internal layers as well as that of the external layers in the case of coated objects, for example paper coated with polyethylene on both sides. In transmission measurements, the IR absorption of the basic material is usually extremely high compared with the water absorption. This frequently leads to intensity difficulties. By contrast, the microwave absorption of dry baryta paper carton 300 μm in thickness is approximately equivalent to a water absorption of 1 to 3% by weight of H_2O.

A high degree of accuracy of the water measurement, e.g. to 0.02 g/m^2 in 5—20 g/m^2, requires a sufficiently high absorption of the incident microwave. Processes which are based on a simple microwave transmission give only low absorptions in this moisture range and when using technically easily available microwave frequencies of up to about 20 GHz in the case of very thin objects in which the electrical thickness $\sqrt{\varepsilon}$ d is very small compared with the wavelength. The use of short microwaves would be a solution but greatly increases the cost of the measuring device.

It is an object of this invention to develop a microwave measuring process which allows continuous determination of small quantities of water in webs of paper or foil to be carried out. It is intended in particular for measuring the residual moisture of photographic paper or film. High standards of accuracy of measurement are required.

According to the invention there is provided a method for the continuous determination of the water content in a moving web, wherein the web is passed through the gap of a two-part resonator in a direction parallel to the electric field component, and the attenuation of the resonator caused by microwave absorption in the web is measured, wherein the resonator is fed from a frequency modulated microwave generator, the frequency modulation amplitude being such that the complete resonance curve both of the empty resonator and of the resonator containing the web is covered, and the change in the Q-factor of the resonator, which is due to the absorption loss in the web, is measured.

The resonator is put out of tune by the web. It is preferred to use a microwave oscillator which has an approximately constant output over the whole range of frequency swept.

Further developments of the invention involve the construction of the resonator, the coupling arrangements and the measurement of small bands.

The most important improvement employed in the apparatus described herein, compared with the processes previously practised, is based on the fact that the microwave is passed several times through the web instead of only once so as to increase the microwave absorption by the water in the web. For this purpose, the web is passed through a suitable microwave resonator which has an adjustable Q-factor

$$(Q=\frac{W_o}{\Delta W}$$

where W_o equals resonance frequency and ΔW equals width at half maximum intensity of resonance amplitude) approximately equal to the number of transmissions. Values between 50 and 5000 can easily be realised and ensure good adaptation to the water contents which may be expected.

Another important property of the process is that only microwaves conducted through hollow metal bodies are used so that there is no radiation and the accuracy of measurement is in no way affected by existing wave fields in the surrounding space.

Contact-free measurments on moving webs can be carried out with two-part microwave resonators and the size of the air gap through which the product is passed can be adapted to the requirements by using suitable types of microwave fields and frequencies. For carrying out measurements on individual samples in the laboratory, e.g. for profile measurements transversely to the web, contact measurement may be employed (width of gap=thickness of web). The results indicated can be made highly insensitive to variations in the width of the web by suitably adapting the wavelength of the tube to the width of the web and suitably orientating the edge of the web in the field minimum. Other forms of resonators may also be used, in which the objects which are to be measured extend beyond the active cross-section of measurement on all sides, so that the results are not influenced by boundary edges.

Physical nature and selectivity of the measuring effect

Sufficient selectivity of the microwave measurement in its response to the water content can usually be obtained by a suitable choice of the measuring frequency. The microwave absorption used for the water measurement is based on the anomalous dispersion of polar molecules in the centimetre wave region. According to the dipole theory (P. Debye, Physik. Zeitschrift (1934) 101/106), an appreciable quantity of time is required for orientation of the electric dipole moments in an alternating electric field at high frequencies. A phase difference therefore exists between the energising field and the polarisation produced, and this causes absorption which is always associated with the anomalous dispersion. These electric losses are particularly marked in highly polar molecules and generally increase with the dipole moment. They show a maximum at a characteristic frequency which is determined by the time required for the dipole molecule to adjust. For the free water molecule, the frequency of the maximum of dipole losses is about 10^{10} Hz (see Figure 1). Restraint of the dipole as a result of being bound to a matrix results in a widening of the absorption curve and shift of the maximum to lower frequencies. Large polar molecules, which are capable of manifesting a competing dipole absorption, have absorption ranges at substantially lower frequencies due to their in most cases higher relaxation times (A. F. Harvey, Microwave Engineering, Academic Press London and New York, 1963, page 233 et. seq.) as well as lower absorptions due to smaller dipole moments. Other absorption processes such, for example, as phonon stimulation (E. Amrhein, Ber. Bunsenges. Phys. Chem. 74 (1970) 8/9 807) in polar and non-polar structures (e.g. polymers) have very much smaller effects.

In contrast to IR measurement which is based on OH-absorption at 1.9 μm, microwave measurement distinguishes between fixed (chemisorbed) and loosely bound physically absorbed water. Loosely bound water produces a substantially higher absorption effect (at a correct choice of measuring frequency in accordance with Debye's theory) than fixed water; this manifests itself in an approximately parabolically curved calibration line in the region of low absorptions. Since the important factor in many drying processes is the exact measurement of the loosely bound water, microwave measurement accurately indicates slight changes in the water content even if considerable quantities of fixed water are present.

Compared with the Karl-Fischer method of water determination which has up to now been mainly used for laboratory purposes, which takes at least 5 to 10 minutes per sample, microwave measurement gives the desired result within seconds.

The invention will now be described in more detail with reference to an example illustrated in the drawing.

Figure 1 shows the dielectric constant of water as function of the frequency;

Figure 2 shows the measuring arrangement;

Figure 3 shows a two-part resonator;

Figure 4 is a block circuit diagram of a transmission circuit;

Figure 5 is a block circuit diagram of a reflection circuit; and

Figure 6 shows the resonance curves of the empty resonator and of the resonator containing a web.

The dependence of dielectric constant on frequency has already been described. Since the maximum of the dipole losses for the free water molecule occurs at about 10^{10} Hz

$$\left(\text{curve } \frac{\varepsilon''}{\varepsilon 0} \text{ in Figure 1}\right),$$

the frequency of the microwave generator is selected from this range in order to obtain as high a measuring effect as possible.

Figure 2 shows the principle of measuring the loss using a two-part resonator. Resonator 1 consists of two pieces of rectangular hollow conductor 1a, 1b separated by a gap 2. A web of paper or foil 3 which is to be investigated is passed through the gap 2 parallel to the electric field component in the resonator. It is important to ensure precise movement of the web within the gap 2. The resonator is of the TE_{10n} field type. It is important that the gap 2 should extend along a neutral line of the wall currents because otherwise severe damping would occur. Cylindrical rollers are used in known manner to guide the web 3.

The resonator 1 is connected to a microwave generator at the input end and to a detector at the output end by iris couplings 4. The degree of coupling depends on the diameter and depth of the iris apertures and influences the quality factor of the resonator. The sensitivity of measurement can therefore be adjusted by the geometry of the iris couplings 4. To prevent interference by reflected signals, the microwave conductors leading to the resonator are made free from reflection.

Measurements are generally carried out without contact with the web i.e. the width of the gap is greater than the thickness of the web of paper or foil 3. In special cases, e.g. when measuring materials with insensitive surfaces, contact measurement may be employed. The two halves 1a, 1b of the resonator are then in contact with the two sides of the web.

The edges of the web usually extend beyond the cross-section of the gap so that no interfering edge effects are produced. When measuring narrow webs having a width less than the longitudinal dimension of the resonator, interference can be avoided by adapting the wavelength or a multiple of half the wavelength in the resonator to the width of the web. In addition, the webs must be introduced in such a way that their edges lie on neutral lines of the electric field. Small variations in the width of the web or changes in length of the web will then have no significant effect on the measurement result.

In the embodiment shown in Figure 3, the resonator widens in the form of a cone to the gap. When this form of resonator, the electric microwave field component remains approximately constant in the region of the separating gap. Movements of the web perpendicular to its direction of transport in this case produce only insignificant changes in the measurement result.

Instead of a rectangular resonator, a cylindrical resonator with purely circular polarisation (TE_{01n} field type) may be used. In this case, the web is passed through the resonator in the plane of the electric field at the point of maximum field intensity. This modified arrangement has the advantage of avoiding anisotropic effects of microwave absorption due to orientated structures in the sample, for example orientated paper fibres in a paper web. In circular polarisation, measurement is carried out in all directions.

The microwave measuring arrangement will now be explained with the aid of the basic circuit diagrams (Figure 4 and Figure 5). Figure 4 shows a transmission circuit. The transmission resonator 1 is fed from a microwave oscillator 6 by way of a variable damping element 5. The microwave oscillator used is a varactor modulated Gunn diode oscillator which is frequency modulated by a saw tooth generator 7 in such a way that the relationship between frequency and time is linear. The modulation frequency is about 2 KHz. The choice of a relatively high modulation frequency has the advantage that it can detect even rapid changes in values. This is important, for example, in the case of sudden changes in moisture content along the web of the foil. In practice, a modulation frequency of between 50 Hz and 5 KHz is sufficient. The frequency sweep of 150 MHz at 9 GHz carrier frequency is such that it will certainly cover the resonance curve of the empty resonator and of the resonator containing a sample to be measured (see Figure 6). The microwave output of the oscillator 6 is transmitted to a reference detector 8 and, by way of the variable damping element 5, to the microwave resonator 1 which is connected in transmission. The transmission signal (resonance curve of resonator) is delivered by a measuring microwave rectifier 10 after rectification of the microwave. It has the form shown in Figure 6. The peak amplitude U_1 decreases with increasing water content, and at the same time the resonance frequency shifts to lower values. The peak amplitudes of the detector signal in the filled and empty resonator are taken to be U_1 and U_0.

A reference signal is branched off by the directional coupler 9 and rectified by the

reference rectifier 8. Reference rectifier 8 and measuring rectifier 10 are peak voltage rectifiers. The rectified peak voltage values U_1 and U_0 are then conducted to a difference amplifier 11. The result is indicated by a measuring instrument 12 or recording device. The bridge circuit shown in Figure 4 (the comparison branch consisting of directional coupler 9 and reference detector 8) has the advantage that variations in the surrounding temperature or in the output of the microwave oscillator 6 have practically no influence on the accuracy of measurement.

If desired, a reflection circuit (Figure 5), may, of course, be used instead of a transmission circuit. In that case, a circulator 13 is connected in front of the reflection resonator 1 to conduct the signal reflected by the resonator to the measuring rectifier 10. For small changes in damping by the dielectric loss of the water in the sample, the water content C_{H_2O} may be determined according to the formula

$$C_{H_2O}=k\varepsilon''=f[\delta(\frac{1}{Q})]$$

where

$$\delta(\frac{1}{Q})=1/Q_1-1/Q_0$$

(ASTM standards 13 (1964) 465, W. Eckhardt et al, Zs. angew. Physik 6 (1954) 236). The symbols have the following meanings:

ε'': the imaginary part of the dielectric constant of water
Q_1: the Q-factor of the filled resonator
Q_0: the Q-factor of the empty resonator.

The water content is thus an unambiguous function f of the resonator Q-factor. For absolute measurement, this function must be calibrated. For this purpose, samples with known water contents are introduced into the gap of the resonator.

WHAT WE CLAIM IS:—

1. A method for the continuous determination of the water content in a moving web, wherein the web is passed through the gap of a two-part resonator in a direction parallel to the electric field component, and the attenuation of the resonator caused by microwave absorption in the web is measured, wherein the resonator is fed from a frequency modulated microwave generator, the frequency modulation amplitude being such that the complete resonance curve both of the empty resonator and of the resonator containing the web is covered, and the change in the Q-factor of the resonator, which is due to the absorption loss in the web, is measured.

2. A process as claimed in claim 1, wherein the peak amplitude of the rectified output of the resonator is used for measuring the Q-factor.

3. A process as claimed in claim 2, wherein the output of the resonator is connected to a peak voltage rectifying circuit which gives an output signal which is proportional to the Q-factor.

4. A process as claimed in any preceding claim, wherein a frequency-modulated microwave generator which has an approximately constant output over its frequency range is used.

5. A process as claimed in any preceding claim, wherein the frequency of the frequency modulation is in the range of from 50 Hz to 5 KHz.

6. A process as claimed in any preceding claim, wherein the measuring resonator is a TE_{10n} field type rectangular resonator which has a symmetric gap passing partly or completely through it through which the sample is passed, which gap extends along a neutral line of the wall currents and enables the sample to be moved parallel to the electric field in the longitudinal direction of the resonator.

7. A process as claimed in any of claims 1 to 5, wherein the measuring resonator is cylindrical, with purely circular polarisation, the web being passed through the resonator in the plane of the electric field and at the point of maximum electric field intensity.

8. A process as claimed in any preceding claim, wherein the form and width of the gap are so chosen that an approximately constant electric microwave field component is obtained over the range of the gap such that movements of the web perpendicularly to its surface cause only insignificant changes in the measured results.

9. A process as claimed in any preceding claim, wherein an iris coupling is used for connecting the microwave energy to the measuring resonator.

10. A process as claimed in any preceding claim, wherein the Q-factor of the resonator is determined by the geometry of the iris coupling, i.e. the diameter and depth of the iris aperture, to adjust the sensitivity of measurement.

11. A process as claimed in any preceding claim, wherein in order to measure webs which are narrower than the longitudinal dimension of the resonator, the wavelength or a multiple of half the wavelength in the resonator is adjusted to the width of the web

and the web is introduced in such a manner that its edges lie along neutral lines of the electric field so that small variations in the width or position of the web do not significantly affect the measurement result.

ELKINGTON & FIFE,
Chartered Patent Agents,
52—54 High Holborn House,
London WC1V 6SH,
Agents for the Applicants.

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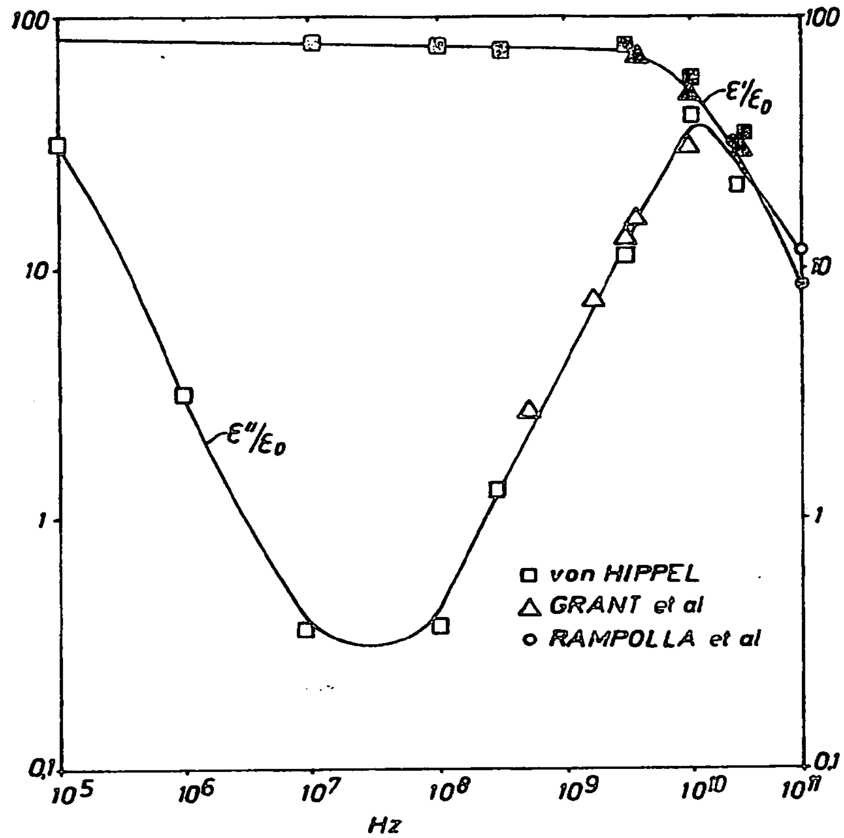
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FIG. 1

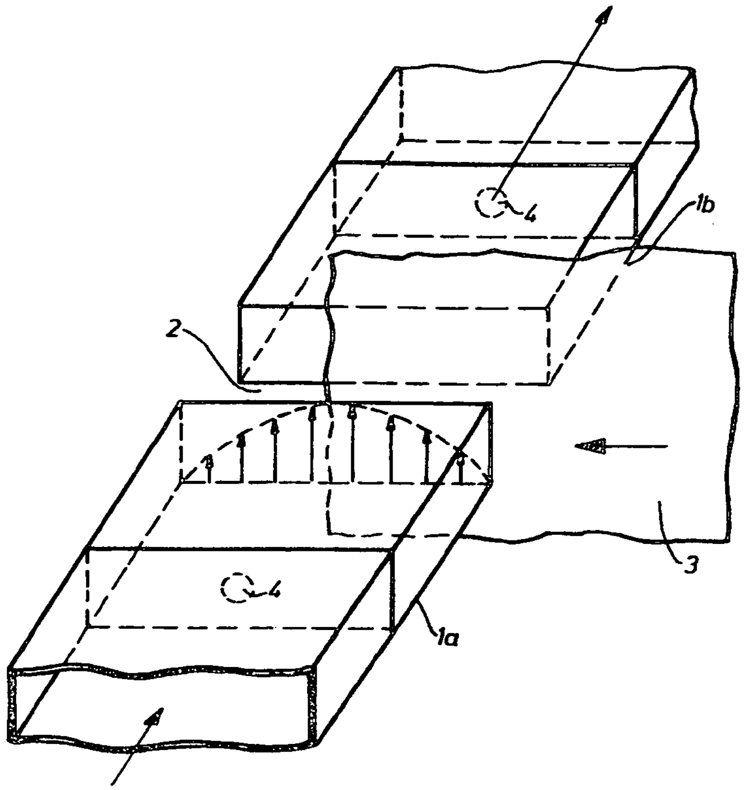

FIG. 2

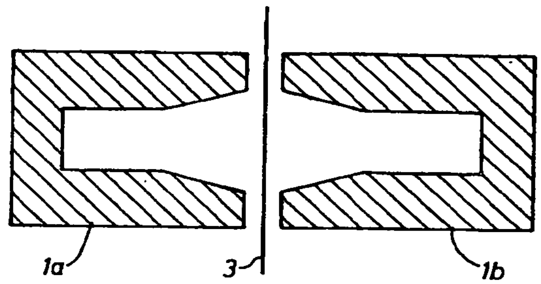

FIG. 3

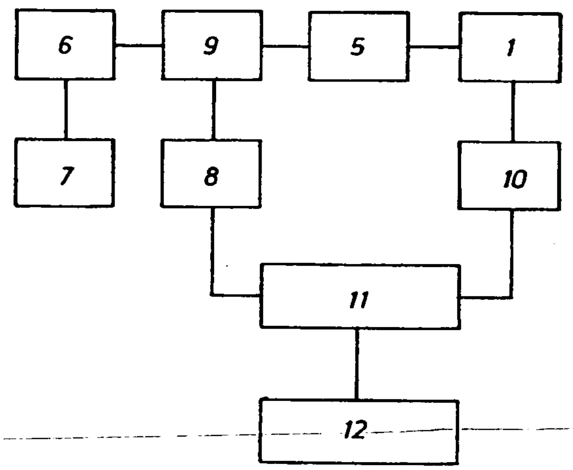

FIG. 4

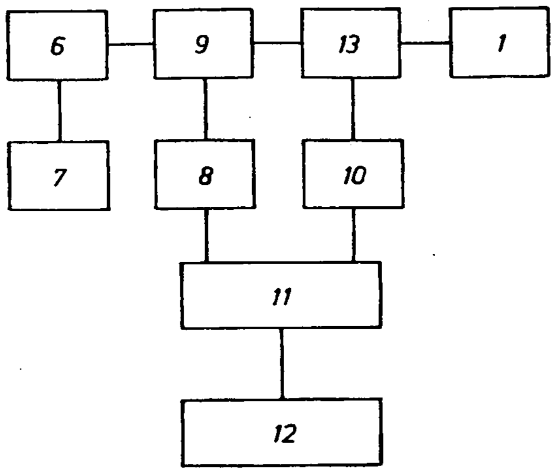

FIG. 5

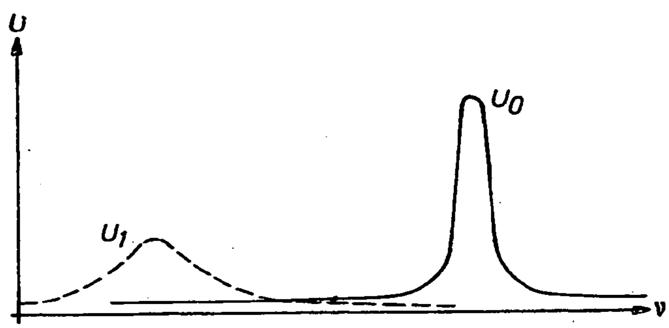

FIG. 6